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# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION.

### Titanium Dioxide Pigment.

We, E. I. DU PONT DE NEMOURS AND COMPANY, a corporation organised and existing under the laws of the State of Delaware, United States of America, of Wilmington, 5 State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in 10 and by the following statement:—

This invention relates to improved titanium dioxide pigments which have high opacity and outstanding ease of dispersibility in liquid coating compositions, such coating 15 compositions, when dried to thin films, having excellent smoothness and outstanding durability upon outdoor exposure. More particularly, the invention is directed to processes for making such pigment compositions.

Titanium dioxide pigments have outstanding properties in paints, enamels and lacquers. Such properties can be further improved by suitable modifications of the 25 pigment particle surfaces as taught in U.S. Patent No. 2,885,366 to Ralph K. Iler, by the surface treatment of calcined titania pigments with a dense skin of silica. The durability of films formed from paints made 30 with such silica-coated pigment is remarkably improved, particularly with regard to outdoor exposure.

To develop the maximum pigment pro-

perties of rutile titanium dioxide made by chloride oxidation processes it is necessary to grind the titanium dioxide at some stage of manufacture. Preferably this is done in fluid energy mills, although ball mills or other conventional mills are sometimes used. In the case of silica-coated titania, if the grinding is done only before the silica treatment, any aggregates formed during the treatment remain in the product and do not disperse in the organic liquids used as the vehicle of paints made therefrom. The gloss of the paint film is adversely affected and has the appearance of containing grit. However, even if the grinding is done after the silica treatment, the desired degree of size reduction is difficult to achieve and the product is difficult to disperse and lacks optimum opacity and gloss in paint films.

The explanation of the above-mentioned difficulties is not readily apparent. Whether there is some affinity between the silica-coated pigment particles which causes them to agglomerate, or whether the grinding of the pigment breaks off some of the silica coating and exposes the titanium particles in a manner such that agglomeration is possible, or whether there is some other explanation, the fact remains that such objectionable lack of gloss and opacity seriously detracts from the utility of the silica-treated pigment.

It has now been determined that the gritty appearance and low opacity is

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associated with a difficult or imperfect dispersibility of the coated pigment.

The present invention is therefore directed to processes for making silica-coated titanium dioxide pigments which have outstanding ease of dispersibility in liquid coating compositions, this ease of dispersibility being indicated by the fact that when pigments treated according to the processes are dispersed in liquid coating compositions, and such compositions are dried to thin films, the films have excellent smoothness and opacity in addition to retaining their outstanding durability. According to the present invention it has been found that if the silica coating is applied to a rutile titanium dioxide pigment in a particular manner, and the so-coated pigment is further treated with alumina formed in a particular manner, the product can be filtered, washed, dried, and ground in fluid energy mills, to give a product having the desired ease of dispersion without sacrifice of durability. The process according to the invention comprises the steps of (1) applying a dense coating of silica to a rutile titanium dioxide pigment made by oxidizing titanium chloride and preferably containing from 0.3 to 3.0% by weight of alumina ( $\text{Al}_2\text{O}_3$ ) formed by co-oxidizing aluminum chloride, the average titanium dioxide particle size being in the range from .1 to 1 micron in diameter, preferably .2 to .5 micron, and the amount of silica being in the range from 0.5 to 25%, preferably 5 to 10%, by weight based on the weight of  $\text{TiO}_2$ , said coating being applied by dispersing the pigment in water and mixing therewith an amount of "active silica", as herein defined, equivalent to the amount of dense silica desired in the final product while maintaining the mixture at a pH above 8, a temperature in the range from 40°C to the boiling point, and an alkali metal ion concentration below 1 normal, preferably below 0.3 normal, said mixing being sufficiently vigorous to substantially prevent precipitation of free silica gel in the mixture, and gradually acidifying the mixture to pH 7 or below in order to precipitate silica as a dense, adherent, amorphous coating on the titanium dioxide particles; (2) mixing with the resultant slurry from 0.5 to 10%, if desired, up to 20 or 25%, by weight of  $\text{Al}_2\text{O}_3$ , based on the  $\text{TiO}_2$ , as an aqueous solution of a water-soluble aluminum compound, while maintaining the pH below 7; (3) adjusting the pH to from 7 to 8 and maintaining the pH in this range until any soluble aluminum present has been precipitated as hydrous aluminum oxide; (4) recovering the product; (5) drying it preferably at a temperature below 450°C and still more preferably below 250°C; and (6) grinding the product to pigmentary size. The preferred method of recovering the product

in step (4) is by filtration and washing to remove at least a substantial portion of the soluble salts formed during the reaction. Spray drying is the preferred method of removing residual water in step (5). Grinding in step (6) is preferably effected by fluid energy means.

The invention is further particularly directed to the pigment produced by the above-described processes. It is characterized as a white, pulverulent composition comprising particles having cores of pigmentary crystalline titanium dioxide, said cores being coated with from 0.5 to 25% of amorphous, dense silica, and there being distributed around said particles from 0.5 to 10% of precipitated alumina, the percentages of silica and alumina being by weight, based on the  $\text{TiO}_2$ . If desired, the amount of alumina can be increased to 20 to 25% to effect further improvement in pigment chalking resistance.

The pigmentary compositions of this invention have a unique combination of properties which give them exceptional utility. When used in conjunction with film-forming materials the films made therefrom have good opacity and outstanding durability upon outdoor exposure. Additionally, the compositions have excellent dispersibility, both in milling operations and during preparation and application of paints containing them.

#### THE CORE MATERIAL.

The titania pigment treated according to this invention is formed by oxidation of titanium chloride at high temperatures, and especially by the co-oxidation of titanium tetrachloride and aluminum chloride, as described, for example, in U.S. Patent No. 2,559,638. The average titanium dioxide particle size should be in the range of 0.05 to .5 micron in radius—that is, the diameter should be from 0.1 to 1.0 micron. Particularly preferred are particles having radii of from .1 to .25 micron. The amount of co-oxidized alumina will ordinarily be in the range of .3 to 3% based on the weight of  $\text{TiO}_2$ . Pigmentary particles having an average size in these ranges are amenable to improvement by the method of this invention. The larger particles are usually aggregates which need to be disintegrated prior to use to get the best results with respect, for example, to good gloss in a paint and hiding power efficiency. For these reasons it is preferred that the primary particles of the base pigment, to which the coating process of this invention is applied, be initially well dispersed.

#### COATING THE CORE PARTICLES WITH SILICA

It is important that the silica coating on the titania be dense rather than porous and

be present as a film or skin around the individual titania particles. The amount of silica present as free silica fragments or gel should be a minimum. Under electron microscope examination at a high degree of magnification, the nature of the coating on the titania particles can be readily seen, because the density of titania is roughly about twice that of silica and the electron beam penetrates the silica more readily than the titania, thus giving a sharp contrast in the density of the image on the electron micrographs. Such micrographs of titania coated with silica by the method described in U.S. Patent No. 2,885,366 clearly show that the silica is present as a coating of substantially uniform thickness around the titania particles. In contrast, in electron micrographs of titania particles with which silica has been precipitated indiscriminately by conventional methods, the silica is present as a voluminous mass of extremely fine particles—that is, a silica gel is formed between the titania particles and not at all thereupon.

U.S. Patent No. 2,885,366 describes various ways in which a dense silica coating can be formed upon various substrates. In an especially practicable method of the present invention the silica is formed upon the titania pigment by slurring the titania in water, adding sufficient ammonia or other suitable basic compound to raise the pH to at least 7, and then adding a desired amount of sodium silicate solution, thereby raising the pH to above 8, preferably about 8 lbs. of silica per 100 lbs. of  $TiO_2$  and thereafter slowly adding dilute sulfuric acid or other dilute acidic compound to form active silica, and continuing acidification until the pH has dropped to 7 or below.

During the silica deposition it is desirable to maintain substantially uniform conditions in the reaction zone to prevent precipitation of free silica gel. This is accomplished by maintaining good agitation and introducing the acid through a distributor designed to avoid local overconcentration of acid. The pH can be followed continuously and should fall gradually as the sodium silicate is neutralized. As the neutralization is completed the slurry can, if desired, be cured for periods such as one hour to permit completion of the deposition of silica onto the surface of the  $TiO_2$  particles.

Stated in greater detail, in the process for coating the core particles titanium dioxide pigment is dispersed in water to form a slurry ranging in concentration from 100 to 700 grams or more of  $TiO_2$  per liter, preferably in the higher concentration range for processing economy. The pH of the slurry is adjusted to above 8 and "active silica" is added. We use the term "active silica" herein in the same sense as is used in U.S. Patent No. 2,885,366. The term is specifically de-

fined from column 6, line 49 to column 7, line 14 in this reference. This definition includes soluble silicic acid prepared by deionizing alkali metal silicate solutions. Silicic acid prepared by the *in situ* reaction of sodium silicate with an acid is the preferred agent.

To this alkaline slurry is then added an acid, typically sulfuric acid, under conditions of good agitation to cause very rapid diffusion of the acid reagent into the alkaline medium, thus substantially avoiding initial local pH conditions below 8. A compatible acidic substance is normally a commercial acid such as sulfuric, nitric, hydrochloric or acetic acid, but any acid substance which does not discolor the product nor react to precipitate an undesired solid may be used. Examples are sodium bisulfate, phosphoric acid, the acid phosphates and carbonic acid. Enough acid is added under these special conditions to precipitate the silica and lower the pH of the slurry to 7 or below.

The coating operation is carried out at an elevated temperature in the range from 40°C to the boiling point, and any alkali metal ion present is maintained at a concentration of less than 1 normal, preferably less than 0.3 normal. Preferred procedures are described in the U.S. Patent No. 2,885,366. The amount of silica in the coating can range from 0.5 to 25% and preferably from 5 to 10% by weight calculated as  $SiO_2$  and based on the  $TiO_2$  content of the product.

Although the final product of this invention exhibits greater surface area per gram of product than does the base pigment, this is believed due in small part to very fine particles of silica dislodged from the complex particles in handling or in milling steps and in large part to the finally precipitated alumina. The basic character of the individual silica-coated particles is believed to resemble quite closely that set forth in the U.S. Patent No. 2,885,366 with respect to surface area parameters. Strict adherence to the conditions of rate of precipitating the silica as set forth in U.S. Patent No. 2,885,366 is not necessary in this instance although that procedure ensures the desired silica coating. It is only essential that a dense, substantially complete coating be obtained by precipitation under alkaline conditions.

Various methods of avoiding localities of low pH during the acidification step can be used. Good agitation and introduction of rather dilute acids are advisable. As a further guard against the creation of pockets of low pH which will have a significant lifetime, the acid solution should be injected and diffused into the alkaline medium in small streams at a multiplicity of points. Agitation and rapid diffusion can be accomplished by recirculation of the slurry through

a pipeline into which series of small streams of acid are fed. A single acid inlet will suffice if the rate of introduction is slow, but a multiplicity of inlets increases the production capacity of the unit. Dilute sulfuric acid in the range of 5 to 25% introduced in small streams has proved satisfactory on a large scale operation.

When the above-described conditions are employed the silica is precipitated on the titania particles present and substantially none of it is deposited as free silica gel not adherent to the pigment particles. The presence of free silica gel can be detected in the final product by means of the electron microscope. Its presence can also be indicated by an increase in the viscosity of the slurry, particularly if such viscosity increases are not uniformly distributed throughout the batch, but rather, are confined to local areas.

The "active silica" used is preferably released from sodium silicate solution or a similar water soluble alkali metal silicate such as potassium silicate. Sodium silicates having a wide range of  $\text{SiO}_2/\text{Na}_2\text{O}$  ratios can be used, but it is preferred that the  $\text{Na}_2\text{O}$  content be high enough to provide a clear water solution for use. Deionized sodium silicate solutions prepared by contacting such solutions with cation exchange resins can be used, thus reducing the soluble salt content of the slurry and greatly lowering the acid requirement. The more or less complete absence of salts, however, may cause difficulty in the subsequent filtration step, but the essential coagulating ions can be introduced in the subsequent step when alumina is added.

#### THE ALUMINA ADDITION

The silica-coated particles in the slurry are next treated in situ with precipitated hydrous alumina in amounts ranging from 0.5 to 10% by weight, calculated as  $\text{Al}_2\text{O}_3$  based on the  $\text{TiO}_2$ . If desired, up to 20 or 25% alumina may be precipitated to effect further improvement in pigment chalking resistance. The preferred amount is in the 2 to 5% range. This is done by adding a soluble aluminum compound to the slurry while maintaining the pH of the slurry below 7 and then adjusting the pH to near neutrality to complete precipitation of the alumina and provide a substantially neutral product.

The precipitation of the alumina must be carried out under the proper conditions. Broadly it is essential to precipitate the alumina from an acid solution of pH below 7, preferably below 5. One procedure for precipitating the hydrous alumina on the acid side is to add aluminum sulfate solution to the slurry while maintaining the pH of the slurry below 3.5, using additional acid if necessary, and then adding a base

to raise the pH to near neutrality, thereby precipitating the alumina. Another procedure employs the simultaneous addition of sodium aluminate solution and an acid or acid salt such as aluminum sulphate in such proportions that the pH is maintained below 6. In a specific case this simultaneous procedure could be carried out at pH 5 whereby most of the alumina is immediately precipitated with the remainder being precipitated upon adjustment of the pH to 7. In all cases it is necessary to adjust the pH of the slurry to near neutrality, i.e. in the range pH 7 to 8 before filtering and washing.

The alumina, thus precipitated, does not necessarily coat the pigment particles but at least forms a separate phase intimately mixed with and distributed around them. Although electron-microscopy does not distinguish between the silica and the alumina, this conclusion is inferred from the appearance of electronmicrographs taken before and after addition of the alumina. If the alumina is added under high pH conditions and then acidified, the outstanding advantages herein described are not obtained.

#### RECOVERING THE PRODUCT

The slurry of  $\text{TiO}_2$  treated by the steps just described has certain characteristic rheological properties. Although very thixotropic it may be picked up and dewatered on the usual vacuum rotary-type filter and washed while still on the filter. If, however, it is removed from the filter and repulped for washing, it is found to be rather unmanageable, being extremely thixotropic. For this reason the filtering and washing are done without removal from the filter. The soluble salts are thus washed out. Washing is preferably continued until the filtrate is quite free of ions. The washed cake is then ready for drying.

#### THE DRYING AND GRINDING STEPS

The preferred commercial method of drying the product is spray drying with either heated air or superheated steam, preferably at temperatures which do not heat the pigment to 450°C or above. Variations in the drying temperature may be employed to give minor control of the ultimate pigment properties.

The dried pigment is ground, preferably in a fluid energy mill, to disintegrate loose aggregates which have formed in the drying.

#### THE PRODUCTS

Electron micrographs of the products prepared as above described show the presence of a uniform dense coating on the  $\text{TiO}_2$ . There is some background debris which is probably loose silica or alumina resulting from the grinding operation. The pigment is readily dispersible in conven-

tional vehicles to give coating compositions having all of the conventional advantages of such compositions pigmented with titania and which have the further advantages that they form smooth, blemish-free films upon draw-down which films have good opacity outstanding durability upon outdoor exposure.

The invention will be better understood by reference to the following illustrative example.

#### EXAMPLE

This example illustrates a method of applying the improvement of this invention to a typical rutile pigment. The base or core material used was prepared by the high temperature oxidation of titanium tetrachloride vapor containing an amount of aluminum trichloride equivalent to 1.2%  $\text{Al}_2\text{O}_3$  based on  $\text{TiO}_2$ . The vapors were mixed in a high temperature reaction zone with oxygen containing a small amount of water vapor and a small amount of potassium chloride, in accordance with techniques described in U.S. Patents Nos. 2,791,490; 2,488,440; 2,559,638 and 3,208,866. The base rutile pigment so produced had a normal particle diameter distribution with maximum frequency of 0.22 micron and surface area of 8.5 square meters per gram.

Forty two thousand pounds of this pigment were slurried in sufficient water to give a concentration of 720 gm/liter. This slurry had a pH of 3.7 due to residual HCl and  $\text{Cl}_2$  from the oxidation step. This slurry was made in a tank having a strong sweep agitator. Finally, sufficient water was added to reduce the  $\text{TiO}_2$  concentration to 300 g/l.

Then, 45 gal. of 28% aqueous ammonia were added to raise the pH to 9.1 and 2,012 gal. of sodium silicate solution containing 200 g/l  $\text{SiO}_2$ , equivalent to 8%  $\text{SiO}_2$  by weight, based on the  $\text{TiO}_2$ , were added during the agitation over a period of about two hours to ensure good mixing and a stable condition of alkalinity. The pH was then 11.0. To further reduce the pigment concentration to 200 g/l 6530 gal. of water were added, and the slurry was heated to 90°C in about one hour. The pH of the slurry was then gradually lowered, and silica was precipitated upon the titania particles, by adding 10% sulfuric acid through a special distributor pipe having  $\frac{1}{4}$ " holes spaced 12" apart mounted horizontally just above the slurry. Addition of 2,060 gal. of acid was effected over a period of five hours. By means of pH electrodes mounted in the tank it was shown that the pH of the slurry was gradually lowered during this period, remaining above 8 until near the end of the acid addition. The pH readings with time were as follows:

Start of acid addition	11.0	
2 hours	10.2	65
3 hours	9.7	
4 hours	8.2	
4½ hours	6.1	

After acid addition the slurry was cured by holding it one more hour with agitation. Then, 296 gal. of sodium aluminate solution containing 2.84 lbs.  $\text{Al}_2\text{O}_3$ /gal., equivalent to 2%  $\text{Al}_2\text{O}_3$  based on the  $\text{TiO}_2$  were added simultaneously with 66°Bé sulfuric acid over a period of  $\frac{1}{2}$  hour, leading with the acid so as to maintain the pH of the slurry at about 3.5, whereby some of the alumina was precipitated in the product. Then 50% NaOH solution was added to adjust the pH to 7, using small increments at the end to equilibrate the system and complete precipitation of the alumina.

The slurry was pumped to rotary vacuum washing filters where it was picked up, washed and dewatered. The washing was adjusted so that the washed cake had a specific electrical resistance of at least 7,000 ohms. The discharged filter cake was repulped, adding a minimum of water when necessary, and fed to a spray dryer. Hot air was used in the spray dryer and the dried pigment reached temperatures of from 125 to 150°C. The dry pigment was ground in a fluid energy mill with steam at 250°C.

This treated pigment contained 3.2%  $\text{Al}_2\text{O}_3$  and 8%  $\text{SiO}_2$  based on the  $\text{TiO}_2$  content. However, when the pigment was leached with 10% sulfuric acid at 50 to 80°C for two hours only 2% of  $\text{Al}_2\text{O}_3$  was removed by solution, the remaining 1.2%  $\text{Al}_2\text{O}_3$  being within the  $\text{TiO}_2$  core. Further leaching with 20% NaOH dissolved silica corresponding to 8% based on the  $\text{TiO}_2$ , as well as about 0.5 more  $\text{Al}_2\text{O}_3$ , presumably from the  $\text{TiO}_2$  core. From leaching tests of this type it was evident that the product comprised a core of  $\text{TiO}_2$  containing 1.2%  $\text{Al}_2\text{O}_3$ , only part of which is available to caustic-leaching. On this core is a dense adherent layer of silica and outside this layer the precipitated alumina is found available to acid-leaching. Electron microscopic studies reveal that this dense  $\text{TiO}_2$  core is surrounded with a more transparent (to electrons) adherent layer of amorphous silica. The precipitated alumina is seen as a loose, fine phase between the coated particles. The surface area of the product was 22 square meters per gram.

The impervious nature of the silica coating obtained by this procedure is shown by its resistance to hot concentrated sulfuric acid. A sample of pigment produced by this example was heated five hours in 96% sulfuric acid at 196°C. Only about .4% of the  $\text{TiO}_2$  was dissolved. As a control another rutile pigment, also silica- and

alumina-treated but not in accord with the method of this invention, was tested in an identical manner. Ninety-five percent of the  $TiO_2$  was dissolved.

5 The pigment of this example shows excellent dispersibility in paint vehicles, and such dispersion can be dried to give films of excellent gloss.

10 Instead of using sodium aluminate as the source of alumina as in the example, the source of alumina can be aluminum sulfate or aluminum chloride. Thus, one can add an aluminum sulfate or chloride solution instead of simultaneously adding sodium aluminate and sulfuric acid.

**WHAT WE CLAIM IS:—**

1. A process for producing rutile titanium dioxide pigment compositions, which comprises (1) applying a dense coating of silica to a rutile titanium dioxide pigment made by oxidising titanium chloride, the average titanium dioxide particle size being in the range from 0.1 to 1 micron in diameter, and the amount of silica being in the range from 0.5 to 25% by weight based on the weight of  $TiO_2$ , said coating being applied by dispersing the pigment in water and mixing therewith an amount of "active silica", as herein defined, equivalent to the amount of dense silica desired in the final product while maintaining the mixture at a pH above 8, at a temperature in the range from 40°C to the boiling point, and at an alkali metal ion concentration below 1 normal, said mixing being sufficiently vigorous to substantially prevent precipitation of free silica gel in the mixture, and gradually acidifying the mixture to pH 7 or below in order to precipitate silica as a dense, adherent, amorphous coating on the titanium dioxide particles; (2) mixing with the resultant slurry from 0.5 to 10% by weight of  $Al_2O_3$ , based on the  $TiO_2$ , as an aqueous solution of a water-soluble aluminum compound while maintaining the pH below 7; (3) adjusting the pH to from 7 to 8 and maintaining it in this range until any soluble aluminum present has been precipitated as hydrous aluminum oxide; and (4) recovering, drying and grinding the product to pigmentary size.

50 2. A process according to claim 1, in

which the rutile titanium dioxide pigment used as the starting material in step (1) contains from 0.3 to 3% by weight of alumina ( $Al_2O_3$ ) formed by co-oxidizing aluminum chloride with titanium chloride and the average titanium dioxide particle size is in the range of 0.2 to 0.5 microns in diameter.

55 3. A process according to claim 1 or 2, in which the amount of silica in the silica coating is in the range of 5 to 10% by weight.

60 4. A process according to any of claims 1 to 3, in which the alkali metal ion concentration is maintained below 0.3 normal during the silica coating step (1).

65 5. A process according to any of claims 1 to 4, in which the slurry is maintained at a pH below 3.5 in step (2) whilst aluminum sulphate is added, together with additional acid if necessary.

70 6. A process according to any of claims 1 to 5, in which the product is dried at a temperature below 250°C.

75 7. A process according to any of claims 1 to 6, in which the active silica is formed *in situ* by adding a solution of sodium silicate, and lowering the pH of the dispersion with dilute sulfuric acid.

80 8. A process for making a titanium dioxide pigment composition as claimed in claim 1, substantially as herein described with reference to the Example.

85 9. A modification of the process claimed in any of claims 1 to 8 in which the amount of alumina is from 0.5 to 25% by weight of  $Al_2O_3$ , based on the  $TiO_2$ .

90 10. A process according to claim 9, in which the amount of alumina is up to 20% by weight of  $Al_2O_3$ , based on the  $TiO_2$ .

95 11. A titanium dioxide pigment composition when prepared by the process as claimed in any of claims 1 to 8.

12. A titanium dioxide pigment composition when prepared by the process as claimed in either claim 9 or 10.

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